



Issues on Fabrication and Evaluation of SiC/SiC Tubes With Various Fiber Architectures

H.M. Yun
Cleveland State University, Cleveland, Ohio

J.A. DiCarlo and D.S. Fox
Glenn Research Center, Cleveland, Ohio

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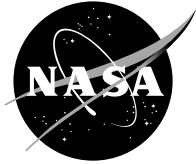
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H.M. Yun
Cleveland State University, Cleveland, Ohio

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Glenn Research Center, Cleveland, Ohio

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ISSUES ON FABRICATION AND EVALUATION OF SiC/SiC TUBES WITH VARIOUS FIBER ARCHITECTURES

H.M. Yun*

Cleveland State University

Cleveland, Ohio 44115

J.A. DiCarlo and D.S. Fox

National Aeronautics and Space Administration

Glenn Research Center

Cleveland, Ohio 44135

ABSTRACT

In order to evaluate potential scale-up issues associated with the fabrication of high-performance complex-shaped SiC/SiC engine components, high-modulus Sylramic-iBN SiC fiber tows were used to form nine different tubular architectural preforms with 13 mm (0.5") inner diameter and lengths of ~ 75 and 230 mm (~3 and ~9"). The thin-walled preforms were then coated with a BN interphase and densified with a hybrid SiC matrix using nearly the same process steps previously established for slurry-cast melt-infiltrated Sylramic-iBN/BN/SiC flat panels. The as-fabricated CMC tubes were microstructurally evaluated and tested for tensile hoop and flexural behavior, and some of the tubes were also tested in a low-pressure burner rig test with a high thru-thickness thermal gradient. To date, four general tube scale-up issues have been identified: (1) greater CVI deposits on outer wall than inner wall, (2) increased ply thickness and reduced fiber fraction, (3) poor test standards for accurately determining the hoop strength of a small-diameter tube, and (4) poor hoop strength for architectures with seams or ply ends. The underlying mechanisms and possible methods for their minimization are discussed.

INTRODUCTION

SiC/SiC composites are excellent candidate materials for high temperature structural applications where the components need to sustain long times in extremely hostile environments, such as those that exist in the hot-sections of advanced gas turbine engines [1] and in reusable space propulsion systems [2]. NASA has demonstrated fabrication of simple-shaped flat SiC/SiC panels that display state-of-the-art thermo-mechanical properties, such as as-fabricated proportional limit and ultimate tensile strengths of ~200 and 500 MPa, respectively; creep-rupture lives up to 1000 hours at 1315°C in air; and the highest thru-thickness thermal conductivity currently available for any SiC/SiC panel [3, 4]. The thin-walled panels were formed by the balanced lay-up of 8 plies of two-dimensional (2D) 0/90 fabric produced by textile weaving of high-modulus high-performance Sylramic-iBN multi-fiber tows [5]. The flat architectural preforms were then chemically vapor infiltrated through the thickness with a BN interphase and a partial SiC matrix of pre-selected volume content. Remaining porosity in the partial matrix was then filled with slurry-cast SiC particulates followed by the high-temperature melt-infiltration (MI) of elemental silicon, resulting in a hybrid SiC matrix with ~2 vol. % inter-tow porosity. NASA is currently scaling up this MI SiC/SiC system by fabrication of more complex shaped hot-section engine components using the same fiber and nearly the same interphase and matrix processes, but different fiber architectures to best conform to the shape and

*NASA Resident Research Associate at Glenn Research Center.

thermostructural requirements of the individual components. The ultimate goal is to establish fabrication guidelines for the production of such SiC/SiC components as turbine vanes and blades, which will be internally cooled by engine compressor air.

For this study, thin-walled small-diameter SiC/SiC tubes with nine different architectures were fabricated as a first step toward gaining a better understanding of potential scale-up issues for complex-shaped components formed from slurry-cast MI SiC/SiC. On the technical side, the CMC tubes are potential sub-elements for cooled leading edges of turbine vanes and blades in gas turbine engines, as well as for structures to provide cooling in advanced space propulsion engines. On the processing side, the tube architectures may introduce addition constituent and process-related issues that are not typically experienced in the fabrication of flat panels, such as increased fiber bending, limited fiber fraction in key directions, and limited interphase and matrix formation caused by one-sided infiltration in contrast to two-sided infiltration for the flat panels. Also, tests that adequately evaluate the thermo-mechanical properties of the tubes are not as straight forward as those established for flat panels. Hence, the objectives of this study were to examine whether these issues can arise for slurry-cast MI SiC/SiC tube fabrication and, if significant, to suggest possible methods for minimizing their effects.

EXPERIMENTAL

Table 1 lists some key properties of the nine fiber architectures used in this study to fabricate slurry-cast MI SiC/SiC tubes with 13 mm (0.5") inner diameter. The first and second columns list the method of construction and a key characteristic of each architecture. All architectures, including the five harness satin (5HS) 0/90 fabric for the jelly roll, were produced at Albany International Techniweave Inc. (AITI, Rochester, NH) by textile forming Sylramic fiber tow onto 30 cm. long graphite tools with 0.5 in. outer-diameter. The tooled preforms were then sent to NASA for conversion into Sylramic-iBN using processes previously established for flat panel architectures [3]. The remaining columns of Table 1 provide data on tow angle from the hoop direction, the number

Table 1. Key Properties of the Nine Sylramic-iBN Fiber Architectures for this study

<i>Fiber Architecture</i>	<i>Major Goal</i>	<i>Fiber angles from hoop</i>	<i># of Splices</i>	<i>Fiber vol. %*</i>	<i># of layers</i>
<i>Braid, bi-axial</i>	Equal braid angle	± 45	0	29<21>	5
<i>Braid, bi-axial</i>	Highest braid angle	± 25	0	31<28>	7
<i>Braid, tri-axial</i>	Equal braid angle	± 30	0	28<24>	7
<i>Braid, tri-axial</i>	Highest braid angle	± 23	0	29<25>	6
<i>Pin-Weave, 2-float</i>	High fiber bending	1	0	32<27>	3
<i>Pin-Weave, 3-float</i>	Low fiber bending	1	0	33<28>	3
<i>Jelly-Roll</i>	18epi 0/90-5HS	0	2	36<18>	6
<i>Filament-Winding</i>	Straight & well aligned hoop fibers	$2/\pm 30$	0	31<28>	9
<i>Orthogonal-Weave</i>	Pin-weave + small thru-layer fibers	3	0	27<14>	6

- Total fiber volume fraction <equivalent fiber volume fraction in the hoop direction>

of ply ends within the total preform, total fiber fraction and equivalent in the hoop direction, and number of repeatable layers through the preform thickness. Two sets of preforms, 75 and 230 mm long, were then prepared and sent to GE Power Systems Composites (GEPSC, Newark, DE) for formation of a CVI-BN interphase coating and CVI-SiC matrix with >30% residual porosity using standard panel processing steps. However, for the CVI BN process, infiltration was one sided through the outer wall of the preforms. For the CVI SiC, infiltration was through both the outer and inner walls, but gas conditions within the tube were not enhanced to account for the preform small diameter and long length. Final SiC/SiC tubes from these preforms were produced at GEPSC by standard two-sided filling of the open porosity with SiC slurry plus melt-infiltrated silicon.

For microstructural and hoop strength evaluation, ring-shaped specimens with ~5 mm width were machined near the top and bottom of each SiC/SiC tube and at the middle of the 230 mm tubes. These ring specimens were prepared both after the CVI-SiC infiltration and after the final MI. After diamond grinding to minimize machining related surface flaws, the hoop-strength specimens were subjected to the Split "D" tensile test (ASTM D2290-00) because the typical tube burst test was not applicable due to the small tube diameter, the high pressure (>25MPa) required for CMC fracture, and the difficult sealing requirement at the specimen ends. Using >3 specimens, an average hoop strength for each tube was calculated using Lame's equation [6] and assuming a thick wall tube without a bending moment. Test specimens of ~5 mm length were also utilized to measure their average flexural strength (ASTM C1161) by loading at two locations that are situated each other a half circle away of the ring assuming bending in the curved beam [7]. In addition, test specimens of ~150 mm length were also machined from the 230mm tubes, and exposed to the NASA mach 0.3 atmospheric pressure burner rig (LPBR) for ~100 hours by impinging combustion gas on the tube outer wall and cooling the inner wall by flowing cold air at a constant rate. The hot and cold side temperatures were monitored, respectively, by an optical pyrometer and type K thermocouple in contact with the inside wall. Post-exposure NDE of the tubes was conducted utilizing x-ray to identify any damaged locations.

RESULTS AND DISCUSSION

Microstructural Evaluation of Tubes

For all architectures, because the CVI processes were primarily through the outer wall, there appeared to exist a tendency for a thicker BN coating and more CVI-SiC infiltration on the tube outside wall in comparison to the inside wall. Nevertheless, the thicknesses of both constituents on the inside wall were within the acceptable range established for the flat panels. In addition, architectural effects on the CVI SiC and slurry/MI processes were also detected based on weight gain measurements that correlated well with microstructural observations. Table 2 indicates these effects as deviations from the average volume fraction values of $X \approx 20\%$ for the CVI SiC and $Y \approx 40\%$ for the slurry/Si. For example, in comparison to the other architectures, the tri-axial braid and the jelly-rolled showed a higher CVI-SiC infiltration, while the 3-float pin weave and the bi-axial braid showed a higher slurry/MI infiltration. Except for the bi-axial braid, total fiber volume fractions in the final SiC/SiC tubes were in the same range of ~30% (Table 1). However, even though the architectures were complex and the tube lengths different, the last column in Table 2 shows matrix infiltration indices of >0.9 were obtained, which is only slightly less than those observed for flat panels fabricated by two-sided matrix infiltration. In addition, the tube architectures were typically more complex than the panel architectures with larger inter-tow

volumes that are not conducive to efficient filling by CVI or slurry. These volumes were also greater due the fact that a tool to support and/or compress the outer preform wall during CVI SiC was not used, giving rise to an average ply thickness of ~ 0.30 mm (~ 0.012 "), which is $\sim 20\%$ greater than ply thickness in a flat panels with an equivalent number of plies. . Although this situation should enhance matrix infiltration and subsequent matrix-controlled properties, it also leads to lower total and direction fiber volume fractions. Finally, Table 2 shows that some of the tubes were not perfectly straight due to a lack of a support tool for the outer wall. Alternate tooling approaches should remedy this issue.

Table 2. Microstructural Observations of Matrix Infiltration into the Tube Preforms

<i>Fiber Architecture</i>	<i>CVI-SiC Vol. %, X $\sim 20\%$</i>		<i>Slurry SiC – MI Si Vol. %, Y $\sim 40\%$</i>		<i>Total Fiber vol. %</i>		<i>Slurry – MI Infiltration Index*</i>		<i>Wall Thickness</i>	<i>Tube Straightness</i>
	<i>3" Tube</i>	<i>9" Tube</i>	<i>3"</i>	<i>9"</i>	<i>3"</i>	<i>9"</i>	<i>3"</i>	<i>9"</i>	<i>9"</i>	<i>9"</i>
<i>Braid, bi-axial</i>	$\sim X$	$\sim X$	$Y++^{\#}$	$Y+$	24	24	>0.9	~ 0.9	70	$\sim 3^{\circ}$ off from the axial
<i>Braid, bi-axial (M)+</i>		<i>X++</i>		<i>Y---</i>		<i>35</i>		<i>~0.8</i>	<i>75</i>	$\sim 5^{\circ}$ off from the axial
<i>Braid, tri-axial</i>	<i>X++</i>	<i>X++</i>	$\sim Y$	$Y-$	26	29	$>>0.9$	>0.9	75	straight
<i>Braid, tri-axial (M)+</i>	<i>X++</i>	<i>X++</i>	$\sim Y$	$Y-$	<i>26</i>	<i>31</i>	$>>0.9$	<i>~0.9</i>	<i>70</i>	straight
<i>Pin-Weave, 2-float</i>	$\sim X$	$\sim X$	$Y-$	$Y-$	33	34	<i>~0.8</i>	<i>~0.9</i>	<i>65</i>	$\sim 2^{\circ}$ off from the axial
<i>Pin-Weave, 3-float</i>	$\sim X$	$\sim X$	$Y+$	$Y+$	29	28	$>>0.9$	$>>0.9$	70	$\sim 5^{\circ}$ off from the axial
<i>Jelly-Roll</i>	<i>X++</i>		$Y-$		31		>0.9		65	
<i>Filament-Winding</i>	<i>X+</i>								~ 70	
<i>Orthogonal-Weave</i>	$\sim X$								~ 100	

* Calculated based on the available porosity before slurry MI and after the MI

X-ray examination of all tubes further confirmed good matrix infiltration for both the 75 and 230 mm preforms with no significant fiber breakage, both after CVI-SiC infiltration and after final slurry/MI. Fig. 1 shows typical x-ray radio-graphs of the 75 mm-long slurry/MI tubes. The characteristics of each fiber architecture are clearly visible, including the off-axis direction of the circumferential tows. For the braided tubes, the measured off-axis angles

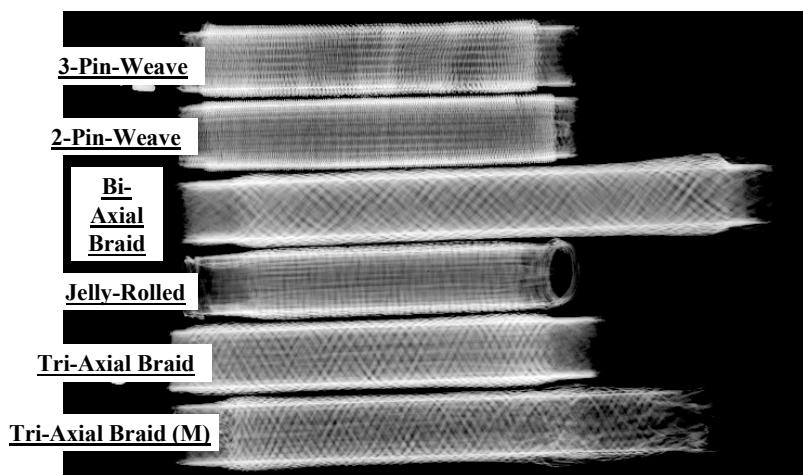


Fig. 1 Typical X-ray radio-graphs of 3" x 0.5" ID Syl-iBN SiC / SiC Tubes after Full Matrix Infiltration

were in good agreement with $<2^\circ$ deviation from the pre-designed braid angles (Table 1), indicating little distortion during composite processing. However, some areas in the 3-float pin weave showed non-homogeneous architectures along the tube length, i.e., tows up to 13° off from hoop direction and tows in densely packed regions. Interestingly, the jelly-rolled tubes did not show a fiber discontinuity, but showed 0/90 fiber distortions in some areas, suggesting that these issues originated during preform handling prior to matrix densification. As expected from Table 1, the tubes with pin-weave and bi-axial braid with the highest braid angles showed denser tow areas than the rest of fiber architectures.

Microstructural examinations on the cross sections of all tubes indicated a uniform interphase and matrix infiltrations for both 3" and 9" length along the entire length. Fig. 2 showed typical microstructures of the tri-axial braid tube with maximum braid angles in the middle

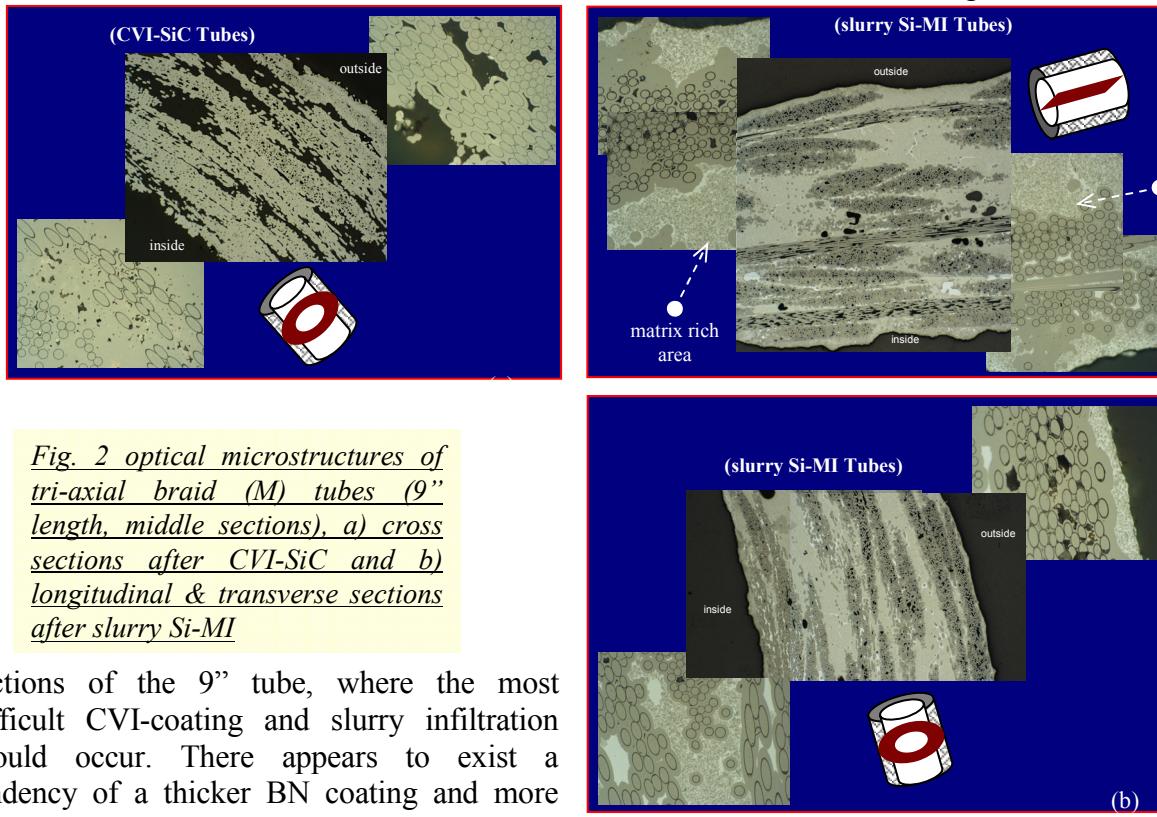


Fig. 2 optical microstructures of tri-axial braid (M) tubes (9" length, middle sections), a) cross sections after CVI-SiC and b) longitudinal & transverse sections after slurry Si-MI

sections of the 9" tube, where the most difficult CVI-coating and slurry infiltration should occur. There appears to exist a tendency of a thicker BN coating and more CVI-SiC infiltration on the tube outside than inside. Nevertheless, the thickness of both constituents on the tube inside was within the range of the flat panels.

Noticeably, there existed a non-uniform fiber loading, and resulted in a slurry Si-MI matrix rich zone, as shown near outside on the longitudinal sections and middle portion on the cross sections in Fig. 2. It was not clear whether this was caused due to fiber architectural imperfections during braiding operation or this was simply because of a complexity of inter-nested braid-layers. Those layer-to-layer inter-nestings and the angled circumferential direction tows within the small diameter tube shaped a variety of cross section tow-views and tow-sizes, and suggested to have a different fiber loading characteristics during structural use than the flat panels, where the tow shape and size are uniform in entire gauge cross sections.

Tube Strength Measurements

Five-millimeter-wide ring specimens from each CVI and MI tube type were tensile loaded to fracture using a "D"-shaped fixture and the same loading rate of 0.13mm/min established for

tensile coupon specimens machined from flat panels. The Fig. 2 load-displacement curves show a monotonic increase in load for all tests, a behavior different than the bi-linear curves typically observed for the panel specimens. This behavior suggests that when tube fracture occurred, the fibers were not able to sustain the load. In support of this argument, the calculated tensile stresses for tube fracture were close to those seen for matrix cracking in panel specimens before and after slurry/MI (~20ksi (~138MPa)). This suggests that the D-test is not adequate for small-diameter CMC tubes, perhaps due to bending stresses that arise after matrix cracking. Furthermore, when the 150mm-long tubes were tested in flexure, the fracture strengths were close to the flex strengths of test bars from flat panels (~80ksi (~550MPa)).

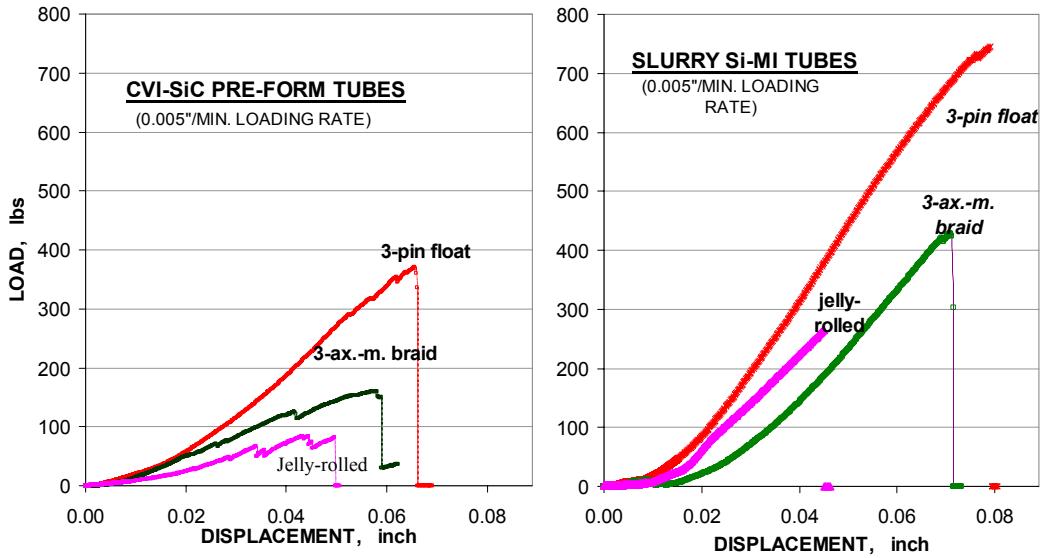


Fig. 3 Typical load vs. displacement curves during the split "D" test on 0.5" ID SiC/SiC tubes, a) after CVI-SiC and b) after slurry Si-MI

Table 3 Summary of failure loads of the tested tubes

Table 3 summarizes all these results for the various tubes after CVI SiC and after slurry/MI, both for the 230mm-long tubes and 75mm-long tubes (data in parenthesis). In this table it can be seen that the D-test strengths increased with higher fiber fraction in the tube hoop direction (with 3-float pin being the

<u>Fiber Architecture</u>	<u>Split "D" failure loads after CVI-SiC, lbs</u>	<u>Split "D" failure loads after MI, lbs</u>	<u>MI-Tube strength (ass. no bending), ksi</u>	<u>MI-Tube flexural strength (ass. w/ bending), ksi</u>
Braid, bi-axial	99(106)*	255(288)	10(11)	60(68)
Braid, bi-axial (M)		299	13	81
Braid, tri-axial	265(175)	415(424)	13(15)	96(100)
Braid, tri-axial (M)	276(141)	480(482)	17(17)	17(114)
Pin-Weave, 2-float	(300)	269(347)	10(13)	64(82)
Pin-Weave, 3-float	(297)	632(640)	22(23)	148(151)
Jelly-Roll	(99)	(216)	(8)	(51)
Filament-Winding		490	18	116

highest), and the flex strengths increased with the highest fiber fraction in the tube

* Maximum failure loads of 9" and (3") length tubes, average of >3 ring specimens adjusted with 4.8mm width by 1.9mm wall thickness. 1000lbs = 4448N, 100ksi = 689MPa

axial direction. Also the D and flex strengths for the jelly-roll architecture were the lowest of all the architectures. This can be attributed to stress risers from the two ply ends or splices in these tube types which have been shown to decrease strength for larger 4" (~102mm) - diameter tubes tested using the standard burst methods [8]. Thus, until better tests are developed for strength testing of small-diameter CMC tubes, it is concluded that little knockdown in fiber strength occurred during the multiple processes for slurry-cast MI SiC/SiC tube fabrication.

Effects of Burner Rig exposure

After exposure for ~100 hours in the NASA low-pressure burner rig with an outer wall temperature of ~1350°C (Fig. 3), x-ray examination of the slurry-cast MI SiC/SiC tubes showed no internal damage relative to as-fabricated tubes. This was the case even though there existed a thermal gradient of ~190°C between the outer wall and the cooled inner wall. Based on $\sigma_{\text{thermal}} \approx E \alpha \Delta T$, this gradient corresponded to a thermal stress of ~25ksi (~170MPa) Microstructural examination and the post-exposure tube strength measurements are planned.

SUMMARY AND CONCLUSIONS

Thin-walled small-diameter (13 mm ID) SiC/SiC tubes of length 75 and 230mm were successfully fabricated with nine different fiber architectures using Sylramic-iBN SiC fibers and interphase and matrix processes currently used to produce high-performance slurry-cast MI SiC/SiC flat panels. Microstructural observations showed no significant issues due to one-sided interphase and matrix deposition by CVI, but only slightly thicker deposits on the outer wall and slightly thicker plies. All tubes displayed good slurry/Si infiltration and high density (~2.8 g/cc). Due to bending moments, it was concluded that the split "D" test for evaluation of tube hoop strength was inadequate for measuring ultimate strength, but perhaps useful for evaluating matrix cracking strength. In this regard, the highest D-test strength was achieved using a 3-float pin woven architecture (27ksi = 186MPa), followed by filament winding and a tri-axial braid architecture. Since flexural testing of the tubes provided strength values in better agreement with the flex strength of test bars from flat panels, it was concluded that the tube fabrication processes had little effect if any on Sylramic-iBN fiber strength. Burner rig exposure of selected tube types at 1350°C for ~100 hours with a thru-thickness gradient of ~190°C had no apparent effect on the inner structure of the tubes, but microstructural and mechanical testing are continuing.

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Fig. 4 Thermal loading on the ~150mm length tube specimens using NASA LPBR combustion gas environment, hot side temperature was ~1350C and ΔT of ~190C.

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